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IS 6609-1 (1972): Methods of test for commercial blasting explosives and accessories, Part I: Gun powder [CHD 26: Explosives and Pyrotechnics]



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“पुनर्विज्ञापित १९९०”
“RE-AFFIRMED 1990”
IS : 6609 (Part I) - 1972
(Reaffirmed 1984)

Indian Standard “पुनर्विज्ञापित १९९०”
METHODS OF TEST FOR “RE-AFFIRMED 1990”
COMMERCIAL BLASTING EXPLOSIVES
AND ACCESSORIES
PART I GUN POWDER

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August 1973

Indian Standard

METHODS OF TEST FOR COMMERCIAL BLASTING EXPLOSIVES AND ACCESSORIES

PART I GUN POWDER

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Indian Standard
**METHODS OF TEST FOR
COMMERCIAL BLASTING EXPLOSIVES
AND ACCESSORIES**

PART I GUN POWDER

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 23 November 1972, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 The explosives industry in India was for a considerable time confined only to the ordnance factories. However, during the last two decades other manufacturing units have also come into existence. It was, therefore, considered necessary to formulate specifications for various explosives and explosive accessories produced by the industry. Before laying down such specifications, it was felt necessary to prepare standard methods of test for the explosives and the accessories.

0.3 Testing of commercial explosives is of utmost importance for ensuring their safety during transport and handling, stability in storage, and adequate life and performance under all conditions of use. Test methods included in this standard cover these aspects for all the groups or explosives under consideration.

0.4 Depending upon the origin/production of the explosive stores, a large number of test methods are in vogue, which though similar in nature, differ in minor details of experimental procedure and expression/interpretation of results.

0.5 It is a difficult task to select a particular method as the best one. Therefore, choice has been effected in favour of those methods, in which sufficient experience has been gained and where sufficient experimental data are available and which are mutually acceptable to all concerned, namely, the producers, the inspectors and the consumers in the field.

0.6 The test methods cover both general and permitted explosives and accessories, such as detonators, detonating fuses and safety fuses. All items are not covered but only those have been included which are currently used and manufactured in the country. The test methods are covered in the following five parts of this standard:

Part I Gun powder

Part IIA	Explosives, general
Part IIB	Explosives, permitted
Part III	Detonators, general, and permitted
Part IV	Detonating fuses
Part V	Safety fuses

0.7 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes the methods of test for gun powder commonly known as black powder.

2. TEST METHODS

2.1 Quality of Reagents

2.1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960†) shall be used in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

2.2 Safety Precautions — Gun powder is extremely liable to ignition by friction, spark, flame, etc. All precautions should be taken during handling to avoid accidents by friction and by spark. Only non-ferrous implements should be used in handling. Gun powder is also hygroscopic and undue exposure of the sample should be avoided.

2.3 Visual Examination — Examine the gun powder visually for homogeneity and for freedom from lumps, visible impurity and foreign matter.

2.4 Preparation of Samples for Testing

2.4.1 For granulated powder, prepare the sample for tests as follows:

Take sufficient powder to fill a stoppered test tube, 15×2.5 cm. Crush the sample rapidly and coarsely in a hardwood mortar using a boxwood pestle, avoiding undue exposure to the air. It is unnecessary, and also inadvisable to grind the sample finely because of the risk of loss of moisture, and it is sufficient merely to crack open the majority of the

*Rules for rounding off numerical values (revised).

†Specification for water, distilled quality (revised).

grains. Effect the crushing in several small increment, discarding the first and transferring each subsequent increment rapidly to a stoppered test tube. Finally, empty out the contents of the filled tube, mix rapidly and return to the tube.

2.4.2 For mealed powder, no preparation of sample is required.

2.5 Determination of Moisture

2.5.1 Procedure — Weigh rapidly 10 g of the powder, and transfer it to a squat aluminium weighing dish with a close fitting lid. Alternatively, a flat-bottomed glass dish covered with a ground glass disc may be used. Close the dish and weigh it accurately. Lift the lid and expose the powder in an oven at $100 \pm 2^\circ\text{C}$ for 2 hours. Then close the lid, remove the dish to a desiccator for about half an hour to cool, and reweigh it. Repeat the operation till constant mass is obtained.

2.5.2 Calculation

$$\text{Moisture, percent by mass} = 100 \times \frac{M_1 - M_2}{M_1 - M}$$

where

M_1 = mass in g of the material and the dish before heating,

M_2 = mass in g of the material and the dish after heating, and

M = mass in g of the empty dish.

2.6 Determination of Sulphur

2.6.0 General — Sulphur is determined by extraction with carbon disulphide.

2.6.1 Reagent

2.6.1.1 Carbon disulphide

2.6.2 Procedure — Transfer to a tared sintered glass crucible No. G 4 about 5 g of the *prepared sample* and weigh accurately. Extract the sulphur with carbon disulphide either using a suction flask or a continuous extraction Wiley or Soxhlet apparatus till the extraction is complete. Dry the crucible first at 50°C (till the carbon disulphide is driven off) and then at 100°C . Weigh the crucible to constant mass [and keep it for the determination of potassium and sodium nitrates (*see 2.7.1*)]. Calculate the percentage of sulphur on moisture-free sample.

2.6.3 Calculation

$$\text{Sulphur, percent by mass} = \left[\frac{(M_1 - M_2) \times 100}{M} - A \right] \times \frac{100}{100 - A}$$

where

M_1 = mass in g of the crucible and the sample,

M_2 = mass in g of the crucible and the residue after extraction,

M = mass in g of prepared sample taken for the test, and

A = percentage moisture in the sample as determined in 2.5.

2.7 Determination of Potassium Nitrate and Charcoal

2.7.0 General — Potassium and sodium nitrates are determined by water extraction. The quantity of sodium nitrate determined with the help of flame photometer is deducted from it. Charcoal is determined by difference.

2.7.1 Determination of Potassium and Sodium Nitrate

2.7.1.1 Procedure — Pour about 20 ml of warm water over the residue in the crucible obtained in 2.6.2, allow to stand for a few minutes and apply suction by a water pump. Repeat this operation of water extraction until the filtrate is free from nitrate. (No blue colour is produced on addition of 1 or 2 drops of the filtrate to concentrated sulphuric acid containing a few crystals of diphenylamine). Transfer the water extract to a 250-ml volumetric flask and make up the volume to the mark. Pipette out 50-ml portion into a tared dish and evaporate to dryness on a water-bath. Add a few drops of concentrated nitric acid, evaporate to dryness and fuse slightly over a small gas flame. Weigh till constant mass is obtained. Calculate the percentage of nitrates on moisture-free sample. [Retain the fused mass for the determination of total chlorine (see 2.12)].

2.7.1.2 Calculation

Potassium and sodium nitrate, percent by mass

$$= \frac{M_1 - M_2 \times 5 \times 100}{M} \times \frac{100}{100 - A}$$

where

M_1 = mass in g of the dish and the residue,

M_2 = tare in g of the dish,

M = mass in g of the sample taken for test in 2.6.2, and

A = percentage moisture in the sample as determined in 2.5.

2.7.2 Determination of Charcoal — Deduct the mass of sulphur (obtained in 2.6) plus the potassium and sodium nitrate (obtained in 2.7.1) from the mass of the sample to obtain the percentage of charcoal.

2.7.3 Determination of Sodium Nitrate

2.7.3.1 Reagents

a) Solution 1 — Sodium chloride solution — Dissolve 1.271 0 g of freshly

dried sodium chloride, dilute the solution to one litre in a volumetric flask and mix it well. One millilitre of this solution is equivalent to 1.848 mg of sodium nitrate (NaNO_3).

- b) *Solution 2 — Potassium nitrate solution* — Dissolve 100.0 g of the dried potassium nitrate in 750 ml of water. Adjust the temperature of the solution to room temperature, dilute to one litre in a volumetric flask and mix it well. One millilitre of this solution contains 0.10 g of potassium nitrate.
- c) *Standard spray solution* — To calibrate the instrument, prepare the spray solutions by mixing volumes of Solutions 1 and 2, as specified in Table 1 and diluting each mixture to one litre in a volumetric flask. Mix well.

TABLE 1 STANDARD SPRAY SOLUTIONS

SPRAY SOLUTION	SOLUTION 1	SOLUTION 2	SODIUM NITRATE PER 100 ml OF SPRAY SOLUTION
(1)	(2)	(3)	(4)
	(ml)	(ml)	(mg)
Blank	Nil	52.5	Nil
A	2.5	52.5	0.5
B	5.0	52.5	1.0
C	7.5	52.5	1.5
D	12.5	52.5	2.5

2.7.3.2 Procedure

- a) *Calibration of the instrument* — Calibrate the flame photometer using the standard spray solutions according to the procedure given in the directions for the instrument.
- b) *Preparation of the sample solution* — Weigh accurately 7 g of the prepared sample into a sintered glass crucible No. G 4. Support the crucible in a Gooch adaptor fitted to a Buchner flask and extract the sample with 200 ml of hot water using gentle suction. Cool the extract and transfer it to a 250-ml volumetric flask. Dilute the extract to the mark with water and mix it well.
- c) *Determination of sodium in low nitrate fuse powders* — By means of a pipette, transfer 25 ml of the sample solution to a 100-ml volumetric flask. Add 0.7 ml of Solution 2 and dilute to the mark with water. Mix this test solution well. Set the flame photometer using the standard spray Solution D and the blank as described in the

calibration procedure. Spray the test solution and note the galvanometer reading. Repeat it three times and take the mean of the three readings. From the calibration curve read the mass of sodium nitrate present in 100 ml of the sample solution.

Calculation

$$\text{Sodium nitrate, percent by mass} = \frac{\text{mg of NaNO}_3 \text{ per 100 ml}}{7}$$

- d) *Determination of sodium in high nitrate fuse powders* — By means of pipette, transfer 25 ml of the sample solution to a 100-ml volumetric flask, dilute the solution to the mark with water and mix it well. Set the flame photometer and spray the sample solution three times as given in (c) for low nitrate fuse powders.

Calculation

$$\text{Sodium nitrate, percent by mass} = \frac{\text{mg of NaNO}_3 \text{ per 100 ml}}{7}$$

2.8 Flashing Test

2.8.1 Procedure — Take 5 g of the *prepared sample* passing through 1.00-mm IS Sieve and distribute evenly over the bottom of a tared flat bottomed hard glass dish 100 ± 5 mm in diameter and 20 ± 5 mm deep. Place the dish on a fume cupboard and ignite the sample using a length of safety fuse. Observe the flash. Cover the dish with a glass plate, allow it to cool in a desiccator for 15 minutes and weigh. Examine the residue, noting its general state of fineness and the presence of any specks of fused salt or carbon. The occurrence of sparks, if any during flashing shall also be noted.

NOTE — The flashing test should be carried out in the absence of marked air draught.

2.9 Determination of Acidity

2.9.0 General — Two methods are prescribed, namely, Method I and Method II.

2.9.1 Method I

2.9.1.1 Procedure — Treat 5 g of the *prepared sample* with 20 ml of freshly boiled and cooled water which is neutral to congo red paper. Decant the extract through a filter paper and examine the solution for acidity in comparison with a solution of 40 parts per million of sulphuric acid. Test by spotting the liquids upon congo red paper of the grade sensitive to 1 : 25 000 sulphuric acid. Estimate the acidity of the extract by comparing the spots. Report the acidity of the sample to congo red paper as equal to, less than or greater than the standard acid, as the case may be.

2.9.2 Method II

2.9.2.1 Procedure — Treat 5 g of the *prepared sample* with 20 ml of neutral freshly boiled and cooled water. Decant the extract through a filter paper and determine the pH by means of a suitable pH-meter using glass and calomel electrodes.

2.10 Determination of Density

2.10.0 General — Two methods are prescribed, namely (a) density bottle method, and (b) an alternate method using Bianchi's densimeter.

2.10.1 Density Bottle Method

2.10.1.1 Apparatus — as shown in Fig. 1. It consists of a density bottle provided with a two-way stopcock which is connected to an adaptor and also to a vacuum pump.

2.10.1.2 Procedure — Dry the material to a constant mass at 105 to 110°C and place the dried material in a glass-stoppered weighing bottle. Carry out the test in duplicate. Dry the density bottle and its stopper at 105 to 110°C, cool in a desiccator and weigh to the nearest milligram (M_1). Fill the bottle with mercury, and note down the temperature and again weigh (M_2). Empty the bottle and dry again. Place approximately 40 g of the dried material in the dry density bottle and weigh (M_3). Fill the bottle up to one-fourth to one-half of its capacity with mercury by pouring it over the contained sample, and connect to the vacuum pump to remove any adhering air bubbles. Fill it up with mercury and weigh (M_4).

2.10.1.3 Calculation

$$\text{Density} = \frac{(M_3 - M_1) \times d}{[M_2 + (M_3 - M_1)] - M_4}$$

where

M_3 = mass in g of the specific gravity bottle with the material,

M_1 = mass in g of the empty specific gravity bottle

d = density of mercury at test temperature,

M_2 = mass in g of the specific gravity bottle with mercury, and

M_4 = mass in g of the specific gravity bottle with the material and mercury

2.10.2 Alternate Bianchi's Densimeter Method

2.10.2.1 Apparatus — as shown in Fig. 2. It consists of a steel weighing vessel K in the form of a weighing pipette and a glass tube T , to which the former can be attached by means of a screw-in connection. The apparatus when assembled forms a barometer tube, the top of which can be connected

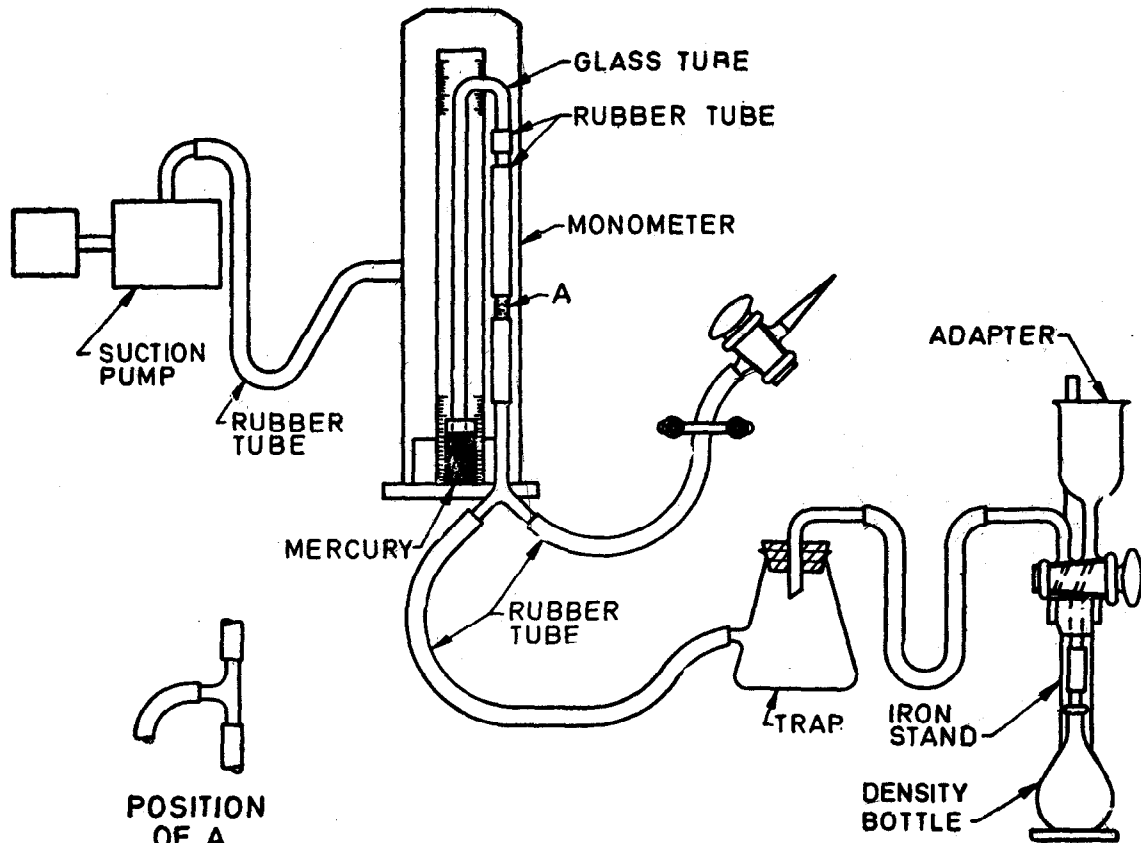


FIG. 1 APPARATUS FOR DETERMINATION OF DENSITY OF GUN POWDER

to a vacuum pump and pressure gauge. The glass tube is fitted with a trap at *A* which prevents any mercury from reaching the pump. A scale is placed behind the glass tube so that the mercury level can easily be read in any particular determination. The taps *C* and *C*₁ of the weighing vessel are detachable, and when in position are screwed to a fixed mark so that the volume of the vessel is kept constant. The apertures at *D* and *E* are fitted with wire gauze strainers to prevent the loss of particles of the powder. The jet below the bottom tap is also detachable. The mercury used in the determination is contained in a trough *B*.

2.10.2.2 Procedure — Place the weighing vessel *K* in position with its lower end dipping about 7.5 cm below the surface of the mercury, the lower stopcock being closed and the upper ones open. Evacuate the apparatus until the pressure is only a few millimetres, shut off the connection to the vacuum pump and gradually open the lower stopcock *C*₁. When the mercury ceases to rise in the tube *A*, close stopcock *C*₁, break the vacuum, close stopcocks *C* and *F*, and disconnect the weighing vessel. Remove the jet, shake any loose mercury from the ends of the weighing vessel, and weigh it full of mercury.

Empty the weighing vessel, insert about 100 g of the gun powder accurately weighed, and repeat the whole procedure, the apparatus being reconnected to the vacuum pump. After the mercury has been allowed to rise in the tube *A*, repeat the operation until no more air can be removed from the interstices of the powder. Finally, obtain the mass of the weighing vessel filled with the mixture of powder and mercury, and calculate the density of the powder.

2.10.2.3 Calculation

$$\text{Density} = \frac{(M_1 \times d)}{(M_2 + M_1) - M_3}$$

where

*M*₁ = mass in g of the material taken for the test,

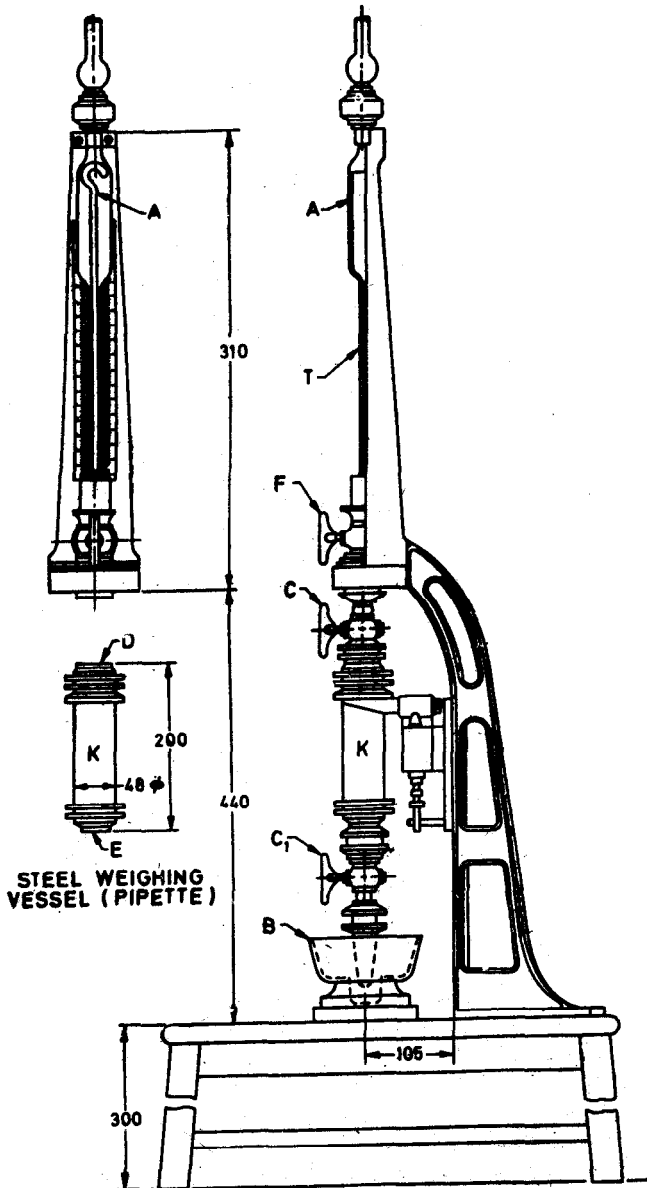
d = density of mercury at test temperature,

*M*₂ = mass in g of the weighing vessel filled with mercury, and

*M*₃ = mass in g of the weighing vessel filled with mercury and powder.

2.11 Determination of Hygroscopicity

2.11.1 Procedure — Weigh accurately, in a covered dish, about 50 g of the material. Transfer it to a 180-micron IS Sieve, 8 cm in internal diameter and 2 cm high, the mesh being of brass or phosphor bronze. Support the sieve on glass rods over a wide shallow dish containing a mush of potassium nitrate, placed in the lower chamber of an incubator, the temperature of



All dimensions in millimetres.

FIG. 2 BIANCHI'S DENSIMETER

which is maintained at $15.5 \pm 2.5^{\circ}\text{C}$ throughout the test. The potassium nitrate mush shall contain a large proportion of the solid phase so that the surface is well broken. Open the inner door of the lower chamber for the minimum time necessary and in such a way that the atmosphere within the chamber is disturbed as little as possible. After 24 hours, transfer the contents of the sieve back to the covered dish and reweigh.

2.11.1.1 Record the percentage gain in mass, plus the percentage moisture content determined as described in 2.5 as the percentage hygroscopicity.

NOTE—Destroy the portion of gun powder which has been used in this test by treating it with water.

2.12 Determination of Total Chlorine (as KClO_4)

2.12.1 Reagents

2.12.1.1 *Dilute nitric acid* — 5 N approximately.

2.12.1.2 *Ether*

2.12.1.3 *Standard silver nitrate solution* — 0.01 N.

2.12.1.4 *Ferric alum indicator* — saturated solution.

2.12.1.5 *Standard ammonium thiocyanate solution* — 0.01 N.

2.12.2 Procedure — Pulverise the dried extracted potassium nitrate as obtained in 2.7.1.1 and transfer it to a hard glass tube 15×4 cm and insert the tube in a hole in a piece of asbestos millboard which has been clamped in an inclined position. Close the tube with a single-bored rubber bung carrying a U-tube containing sufficient water to form a water seal. Heat the potassium nitrate gradually with a Bunsen burner until an active evolution of oxygen is obtained and continue the heating for about 15 minutes. Disconnect the U-tube, allow the test-tube to cool and wash out both the test-tube and the U-tube with water.

2.12.2.1 Transfer the solution so obtained to a 250-ml glass stoppered bottle, acidify with dilute nitric acid, and sufficient ether to form a layer about 1 cm deep on the surface of the liquid. Add silver nitrate solution, 2 ml at a time until no further formation of milkyiness can be detected, the bottle being stoppered after each addition of silver nitrate and shaken well. During the shaking, the silver chloride quickly coagulates and any opalescence due to further precipitation of silver chloride can be easily seen. Add 1 ml of ferric alum indicator and titrate the excess of silver nitrate with ammonium thiocyanate solution with intermittent shaking until a brown coloration appears in the aqueous layer. Carry out a blank experiment on the reagents.

2.12.2.2 The difference of the titres of ammonium thiocyanate is equivalent to the volume of the silver nitrate used and thus to the combined

chloride and perchlorate contents of the powder. Make allowance for the chloride content, determined as in 2.13 and calculate the percentage total chlorine (as KClO_4). If, however, the percentage total chlorine is less than 0.10 percent, there is no need to determine the chloride content separately.

2.12.3 Calculation — Calculate the total chlorine content on the basis that one millilitre of 0.01 N ammonium thiocyanate solution is equivalent to 0.013 9 g of chlorine (as KClO_4).

2.13 Determination of Chlorides

2.13.1 Reagents

2.13.1.1 Standard silver nitrate solution — 0.1 N.

2.13.1.2 Concentrated nitric acid — conforming to IS : 264-1968*

2.13.1.3 Ferric alum indicator — saturated solution.

2.13.1.4 Standard ammonium thiocyanate solution — 0.1 N.

2.13.1.5 Nitrobenzene — freshly vacuum distilled.

2.13.2 Procedure — Weigh accurately about 5 g of the *prepared sample* and extract with water. Filter the residue through a folded filter paper and wash thoroughly with water, collecting the filtrate and washings in 250-ml conical flask. Add with a pipette 25 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 10 ml of nitrobenzene. Shake vigorously and add 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change. Carry out a blank simultaneously with the same quantities of all the reagents.

2.13.3 Calculation

$$\text{Chlorides (as Cl),} \\ \text{percent by mass} = \frac{3.546 (V - V_1) \times N_1}{M} \times \frac{100}{100 - A}$$

where

V = volume in ml of ammonium thiocyanate solution required for the blank titration,

V_1 = volume in ml of ammonium thiocyanate solution required for titration with the material,

N_1 = normality of ammonium thiocyanate solution,

M = mass in g of the sample taken for the test, and

A = percentage of moisture in the sample as determined in 2.5.

*Specification for nitric acid (*first revision*).

2.14 Determination of Chlorates

2.14.1 Reagents

2.14.1.1 Aniline hydrochloride reagent — Dissolve 18 g of the redistilled aniline in 375 ml of concentrated hydrochloric acid and make up the volume to 500 ml with water. Add 2 drops of saturated potassium chlorate solution, shake and allow to stand overnight. Filter off the blue sediment. Store the reagent in a glass stoppered bottle in the dark.

2.14.1.2 Standard potassium chlorate solution — Dissolve 0.2 g of potassium chlorate in water and make up to exactly one litre.

2.14.1.3 Potassium nitrate solution — chlorate-free (10 percent m/v).

2.14.2 Procedure — Extract 5 g of the *prepared sample*, accurately weighed, with 50 ml of warm water, filter, and make up to 100 ml in a Nessler cylinder with the aniline hydrochloride reagent. Compare any blue coloration which develops in 10 minutes with standards prepared from chlorate-free potassium nitrate to which known quantities of the standard potassium chlorate solution and aniline hydrochloride reagent have been added.

NOTE — Commence all the determinations of any one series at the same time since the colour is liable to fade after development. Carry out a preliminary test to ensure that the reagents are effective.

2.15 Determination of Sulphates

2.15.1 Reagents

2.15.1.1 Concentrated hydrochloric acid — see IS : 265-1962*.

2.15.1.2 Barium chloride solution — 10 percent (m/v).

2.15.1.3 Dilute hydrochloric acid — 1 percent (v/v).

2.15.2 Procedure — Weigh accurately about 5 g of the *prepared sample*, transfer it to a beaker and treat with 100 ml of water at 30 to 40°C. Allow the mixture to stand for 15 minutes with occasional stirring. Decant the liquid through Whatman filter paper No. 42 or equivalent, and wash the residue in the beaker twice with cold water, decanting each time as before. Make up the filtrate to about 200 ml with water and acidify with a little concentrated hydrochloric acid. Heat to boiling, add 15 ml of boiling barium chloride solution and boil for further 15 minutes. Allow to stand for 4 hours. Filter the solution containing barium sulphate through a tared sintered glass crucible No. G 4 or on Gooch crucible or a filter paper (Whatman No. 42 or equivalent). Wash with dilute hydrochloric acid till the filtrate is free from barium salts and then with water till it is free from chlorides.

*Specification for hydrochloric acid (revised).

2.15.2.1 If the filtration has been carried out on a sintered glass crucible, dry the precipitate at $110 \pm 5^\circ\text{C}$. In case of Gooch crucible dry the precipitate and ignite it over a burner or in the muffle furnace at 600 to 700°C for half an hour. When the filtration is carried out on a filter paper, ignite the precipitate over a burner or in the muffle furnace at 600 to 700°C for half an hour. After this, cool and add concentrated nitric acid (2 drops) and gently heat to drive off acid fumes. Then ignite again till constant mass is obtained.

2.15.3 Calculation

$$\text{Sulphates (as } \text{SO}_4 \text{), percent by mass} = \frac{41.2 \times M_1}{M}$$

where

M_1 = mass in g of the ignited residue, and

M = mass in g of the sample taken for the test.

NOTE — If the amount of sulphate present is small, the determination may be carried out by comparing the turbidity of the solution against that produced by adding known volumes of 0.01 N sulphuric acid to 200 ml of water and precipitating with 15 ml of barium chloride solution exactly as above.

2.16 Determination of Graphite

2.16.0 Qualitative Test for Graphite — Add 5 g of the prepared sample to about 100 ml of water, stir and allow the bulk of the insoluble matter to settle. Examine the liquid for evidence of graphite on the surface and any indication of glistening particles in the bulk of the liquid. If more than a trace is found in the gun powder, estimate it as described in 2.16.2 and 2.16.4.

2.16.1 Reagents

2.16.1.1 Concentrated nitric acid — See IS : 264-1968*.

2.16.1.2 Concentrated sulphuric acid — See IS : 266-1961†.

2.16.1.3 Potassium nitrate — powdered.

2.16.2 Procedure for Granulated and Mealed Powders — Place 20 g of the prepared sample in a 900-ml beaker, and 120 ml of concentrated nitric acid and heat for half an hour on a sand-bath at $105\text{-}125^\circ\text{C}$. Add cautiously 120 ml of concentrated sulphuric acid, a few ml at a time, since the reaction tends to be vigorous and causes frothing. Continue heating at $105\text{-}125^\circ\text{C}$ for 4 hours. Add further concentrated sulphuric acid, if necessary, to moderate frothing or to wash down scum.

*Specification for nitric acid (first revision).

†Specification for sulphuric acid (revised).

2.16.2.1 Prepare a silica Gooch crucible with asbestos fibre which has been previously digested with a mixture of concentrated nitric acid and sulphuric acid. Wash the prepared crucible thoroughly with water, dry, ignite and weigh. Filter the acid mixture through the crucible with the aid of suction, and wash the insoluble material into the filter with concentrated sulphuric acid. Wash the residue with concentrated sulphuric acid until the filtrate is free from colour. Empty the mixed acids from the Buchner flask, and then wash the crucible thoroughly with water. Dry the crucible to constant mass (M_1) at 103-105°C.

2.16.2.2 The increase in mass is due to the mass of graphite plus inorganic insoluble matter. Ignite the crucible strongly to burn off the graphite, cool the crucible in a desiccator and weigh (M_2) to give, by difference, the graphite content.

2.16.3 Calculation

$$\text{Graphite, percent by mass} = \frac{M_2}{M} \times 100$$

where

M_2 = loss in mass in g after ignition (that is, $= M_1 - M_2$),
and

M = mass in g of the material taken for the test.

2.16.4 Procedure for Pebble Powder — Select and weigh 25 pebbles which are, in shape and size, representative of the whole sample. Let the mass be M . Immerse each pebble, separately, in about 30 ml of hot water in a beaker until the graphited surface appears to be loosened. Remove the pebble by means of tongs and rinse the outer layers into the beaker by means of a jet of hot water. Retain the rinsed pebbles in another beaker.

2.16.4.1 Filter off the graphite-charcoal mixture on a Whatman filter paper No. 42 or equivalent and place the filter paper in a boiling water-oven until it is substantially dry. Transfer the residue from the filter to a tared weighing bottle and dry it to constant mass in an oven at 103-105°C. Let the mass be A .

2.16.4.2 Digest the rinsed pebbles with hot water in a beaker on the boiling water-bath until they are disintegrated. Collect the insoluble matter on a Whatman filter No. 42, dry off most of the moisture as before, transfer the residue to a tared weighing bottle and dry it to constant mass at 103-105°C. Use this material as a blank.

2.16.4.3 Take exactly 2 g of the dried graphite-charcoal mixture from the weighing bottle in 2.16.4.1 and transfer it to a 900-ml beaker. Take 2 g of the other dried residue obtained in 2.16.4.2 in a similar beaker to serve as a blank. To each beaker add 6 g of powdered potassium nitrate, 60 ml of concentrated nitric acid and, finally, 60 ml of concentrated sulphuric

acid, poured gently down the side of the beaker a few ml at a time. The mixture shall be swirled and allowed to stand between successive additions to acid until any froth has subsided. Cover the beakers and heat them on a sand-bath, cautiously at first, to 105-125°C for about 4 hours. The reaction tends to be vigorous and frothing occurs unless great care is taken during the addition of the concentrated sulphuric acid and in the early stages of the heating.

2.16.4.4 Prepare and weigh two Gooch crucibles, using asbestos which has previously been treated with a mixture of concentrated nitric and sulphuric acids. Decant the liquid contents of the beakers separately through the two crucibles, and wash the residues in with concentrated sulphuric acid. Continue to wash the contents of each crucible with concentrated sulphuric acid until the washings are free from colour. Empty and rinse each filter flask and continue washing the crucibles, first with a little cold water and then with hot water until the washings are free from acid. Dry the crucibles to constant mass at 103-105°C. The difference between the mass of the two residue represents the graphite content (G) of the 25 pebbles of powder taken.

2.16.5 Calculation

$$\text{Graphite, percent by mass} = \frac{100 \times AG}{2 M}$$

where

A = total mass in g of charcoal-graphite mixture obtained,

G = mass in g of graphite found, and

M = mass in g of 25 pebbles.

2.17 Determination of Lead Fuse Burning Speed

2.17.0 General — The lead fuse burning speed of a powder provides reliable indication of the probable burning speed of the safety fuse made with that powder. In this test a sample of the powder is used in the preparation of a length of lead fuse and from observed burning speed of the fuse, burning speed under the standard conditions is obtained by applying corrections for temperature, and barometric pressure and moisture content of gun powder.

2.17.1 Apparatus

2.17.1.1 Lead fuse rolling machine — as shown in Fig. 3.

2.17.1.2 Constant temperature bath — maintained at 27°C.

2.17.1.3 Lead tube — The internal diameter of the tubing shall be as nearly as possible 5 mm. It shall be 0.27 to 0.28 kg per metre length. It shall be uniform in wall thickness and circular in section.

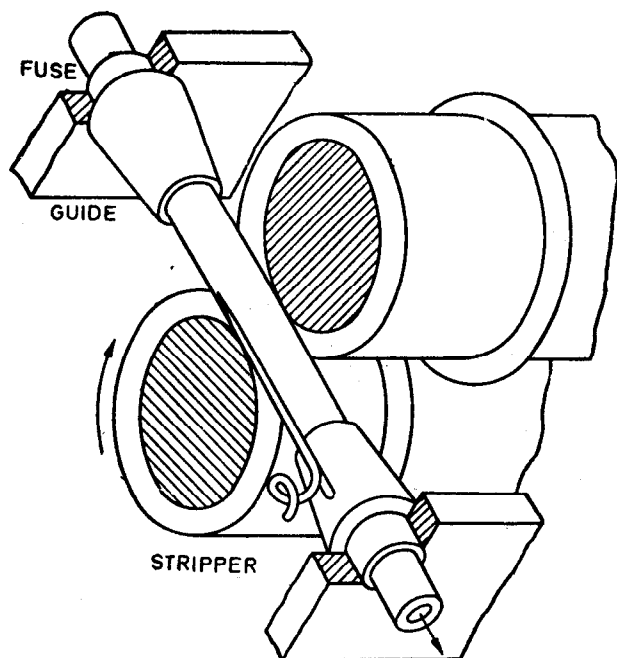


FIG. 3 LEAD FUSE ROLLING APPARATUS

2.17.2 Procedure

2.17.2.1 Preparation of lead fuse

a) *Filling* — Take a lead tube of 41-42 cm length, straighten it and close one end by means of a wooden mallet or with pair of pliers, widen the other end slightly to facilitate filling. Invert the tube and tap it gently to remove any fragments of lead from the bore. Pour the powder into the tube until it is full using a funnel, scoop or other suitable means (10 g of the powder are usually sufficient). Hold the tube vertically and tap it 50 times on a firm base. Add more powder until the tube is again full, and tap it another 50 times. Plug the open end with cotton wool or similar material, and close the lead tube by beating the end with a wooden mallet or by rounding it off with a pair of pliers.

b) *Rolling* — Pass the tube through the seven grooves and corresponding dies of a lead fuse rolling machine, the dies to be used are of dia 6.35, 5.84, 5.33, 4.88, 4.50, 4.22 and 3.99 mm. Lubricate the rolls thoroughly with mineral lubricating oil before commencing the rolling operation and pass the lead tube through the dies in the order of decreasing size. Reverse

the fuse as it comes through each die, so that the last end to come out from one die, is first to enter the next. Rotate the tube through at right angle as it enters each die, so that the fins are not always formed in the same place.

2.17.2.2 Wipe the rolled fuse free from oil and cut a test length of 100 cm from the centre of the sample. Lay the test length in the trough of a water bath maintained at 27°C for not less than 2 minutes; and ignite one end with a fuse or with the spit from another piece of lead fuse. Note to the nearest second by means of a stop watch the time of burning of the test length from the instant of ignition to the final spit. Observe the barometric pressure and if a constant temperature bath is not available, note the room temperature at which the test was carried out.

2.17.3 Corrections — Correct the lead fuse speed for standard conditions of pressure of 76 cm mercury, temperature of 27°C, and percent moisture content of gun powder by the following formulae:

$$T_c = T_0 + 0.09 (t - 27) + 0.91 (p - 76.0) - 5.31 (m - 1.0)$$

where

T_c = corrected time of burning in sec/metre,

T_0 = observed time of burning in sec/metre,

t = room temperature in °C,

p = barometric pressure in cm of mercury, and

m = moisture content of the gun powder percent by mass.

2.18 Determination of Particle Size

2.18.1 Procedure — Assemble the appropriate sieves together with the bottom receiver, the coarser sieve being on the top. Weigh about 100 g of the material and transfer it on the uppermost sieve. Shake the sieves for 15 minutes by hand with frequent tapping at an angle of about 30° to the horizontal on a felt pad, and with occasional rotation in order to ensure distribution of the powder. In case of mealed powder the sieving shall be achieved with the help of a soft camel hair brush.

2.18.1.1 Weigh separately the material passing through each sieve and express it as percentage of the mass of the material taken for the test.

INDIAN STANDARDS

ON

EXPLOSIVES AND RAW MATERIALS FOR EXPLOSIVES

IS:

- 301-1963 Potassium nitrate for explosives and pyrotechnic compositions (*revised*)
- 438-1972 Aluminium powder for explosives and pyrotechnic compositions (*first revision*)
- 708-1970 Potassium chlorate, technical (*revised*)
- 2012-1961 Red phosphorus
- 2307-1962 Magnesium powder for explosives and pyrotechnic compositions
- 4396-1967 Barium nitrate for explosives and pyrotechnic compositions
- 4668-1967 Ammonium nitrate for explosives
- 5670-1970 Lead thiocyanate for explosive and pyrotechnic compositions
- 5671-1970 Strontium nitrate for pyrotechnic compositions
- 5713-1970 Manganese dioxide for explosive and pyrotechnic compositions
- 5731-1970 Antimony sulphide for explosive and pyrotechnic compositions
- 6609 (Part I)-1972 Methods of test for commercial blasting explosives and accessories:
Part I Gun powder
- 6609 (Part IV)-1972 Methods of test for commercial blasting explosives and accessories:
Part IV Detonating fuses
- 6609 (Part V)-1972 Methods of test for commercial blasting explosives and accessories:
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